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IS 6971 (1998): 2-ethyl Hexan-1-OL [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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भारतीय मानक

2-ईथाईल हेक्सान-1-ऑल — विशिष्टि

( पहला पुनरीक्षण )

*Indian Standard*

2-ETHYL HEXAN-1-OL — SPECIFICATION

( *First Revision* )

ICS 71.080.99

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1973. The committee responsible for the formation of this standard decided to revise the same in the light of the experience gained over the years so as to align the same with the latest development in technology. In the present revision, the requirements of two different varieties of the materials have been prescribed depending on its end use in the plasticizer or general industrial purpose.

2-Ethyl Hexan-1-ol [ $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}(\text{C}_2\text{H}_5)\text{CH}_2\text{OH}$ ] is widely used as the raw material for the manufacture of plasticizers. Di-2-Ethyl Hexyl Phthalate (also known as DOP), which is an esterification product of 2-Ethyl Hexan-1-ol and phthalic anhydride, is the most common plasticizer for PVC. Other common phthalate products based on 2-Ethyl Hexan-1-ol include butyl octyl phthalate (BOP) and octyl decyl phthalate. Plasticizers such as trioctyl phosphate, dioctyl adipate and dioctyl sebacate are also based on 2-Ethyl Hexan-1-ol.

2-Ethyl Hexan-1-ol is also used as a chemical intermediate in the manufacture of wetting agents, lacquer solvents and 2-Ethyl Hexyl acetate. It is also used as an excellent defoaming agent and viscosity adjuster in the urea formaldehyde coatings.

In the preparation of this standard considerable assistance has been derived from the following ASTM:

ASTM D 1969 : 1991 Specification for 2-Ethyl Hexan-1-ol

ASTM D 5008 : 1993 Determination of purity

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## Indian Standard

# 2-ETHYL HEXAN-1-OL — SPECIFICATION

( First Revision )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2-Ethyl Hexan-1-ol, suitable for plasticizer manufacture and other industrial applications.

### 2 NORMATIVE REFERENCES

The following Indian Standards are necessary adjuncts to this standard. The standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below :

IS No.	Title
265 : 1993	Hydrochloric acid ( <i>third revision</i> )
266 : 1993	Sulphuric acid ( <i>third revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
2362 : 1994	Determination of water by Karl Fischer method ( <i>first revision</i> )
4161 : 1967	Nessler's cylinder
4905 : 1968	Methods for random sampling
5298 : 1983	Method for determination of distillation range and distillation yield
8768 : 1988	Measurement of colour in liquid chemical products in Hazen units

### 3 GRADES

There shall be two grades of the material, namely:

<i>Plasticizer Grade</i>	intended for use in the plasticizer manufacture and
<i>Industrial Grade</i>	intended for use in other industrial applications.

### 4 REQUIREMENTS

#### 4.1 Description

The material shall be clear and free from matter in suspension and shall consist essentially of 2-Ethyl Hexan-1-ol.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A to G.

**Table 1 Requirements for 2-Ethyl Hexan-1-OL**

Sl No.	Characteristic	Requirements		Methods of Test, Ref to
		Plasticizer Grade (3)	Industrial Grade (4)	
(1)	(2)	(3)	(4)	(5)
i)	Colour, hazen units, <i>Max</i>	10	15	IS 8768
ii)	Relative density, 27°/27°C	0.830 to 0.833	0.830 to 0.833	A
iii)	Distillation range:			
a)	Initial boiling point at 760 mm Hg, <i>Min</i>	180°C	180°C	B
b)	Dry point at 760 mm Hg, <i>Max</i>	186°C	186°C	
iv)	Water content, percent by mass, <i>Max</i>	0.1	0.15	C
v)	Acidity (as acetic acid), percent by mass, <i>Max</i>	0.01	0.02	D
vi)	Aldehydes and ketones (as carbonyl group), percent by mass, <i>Max</i>	0.02	0.05	E
vii)	Sulphuric acid colour hazen units, <i>Max</i>	50	100	F
viii)	Purity, percent by mass	99.0	98.0	G

### 5 PRECAUTION IN HANDLING

2-Ethyl Hexan-1-ol is considered as practically non-toxic but splashes in the eye causing irritation and can result in injury. Eye shields should be worn during handling operations. Splashes in the eye should be removed immediately by washing with copious quantities of water.

### 6 PACKING AND MARKING

#### 6.1 Packing

The materials shall be packed in well-closed containers of mild steel.

#### 6.2 Marking

The container shall be suitably marked with the following information:

- Name of the material;
- Net mass of the material in the container;
- Name of the manufacturer and his recognized trade-mark, if any; and

- d) Batch number or lot number, in code or otherwise.

### 6.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark.

**6.2.2** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 7 SAMPLING

The procedure for sampling and the criteria for conformity of the material shall be prescribed in Annex H.

## 8 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## ANNEX A

[Clause 4.2 and Table 1, Sl No. (ii)]

### RELATIVE DENSITY

#### A-1 OUTLINE OF THE METHOD

In this method mass of equal volumes of the material and water are compared at 27°C.

#### A-2 APPARATUS

**A-2.1 Relative Density Bottle** — 25 ml capacity.

**A-2.2 Water Bath** — maintained at  $27.0 \pm 0.2^\circ\text{C}$ .

**A-2.3 Thermometer** — Any thermometer of a suitable range with  $0.1^\circ\text{C}$  or  $0.2^\circ\text{C}$  subdivisions.

#### A-3 PROCEDURE

Clean and dry the relative density bottle. Weigh and fill with recently boiled and cooled water at 27°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in the water-bath. Keep the entire bulb covered with water and hold at that temperature for 30 minutes. Carefully

remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely, dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

#### A-4 CALCULATION

$$\text{Relative density at } 27^\circ\text{C}/27^\circ\text{C} = \frac{M_1 - M_2}{M_3 - M_2}$$

where

$M_1$  = mass in g of the relative density bottle with the material,

$M_2$  = mass in g of the relative density bottle, and

$M_3$  = mass in g of the relative density bottle with water.

## ANNEX B

[Clause 4.2 and Table 1, Sl No. (iii)]

### DETERMINATION OF BOILING RANGE

#### B-1 PROCEDURE

Determine the boiling range by the procedure as prescribed in IS 5298 applying following corrections.

#### B-2 CORRECTION FOR THERMOMETER READING

##### B-2.1 Error of Scale

In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

##### B-2.2 Correction for Barometric Pressure

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer scale as corrected for error of scale may be used as such. If however the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall also be corrected as follows:

- For every 10 mm above 760 mm Hg, subtract  $0.50^\circ\text{C}$  from the specified temperature; and
- For every 10 mm below 760 mm Hg, add  $0.50^\circ\text{C}$  to the specified temperature.

NOTE — These corrections are valid only for pressure above 700 mm Hg.

**ANNEX C**

[Clause 4.2 and Table 1, Sl No. (iv)]

**DETERMINATION OF MOISTURE CONTENT****C-1 GENERAL**

Moisture is determined by the Karl Fischer method.

**C-2 PROCEDURE**

Take about 20 g of the material, weigh accurately and determine the moisture content by the procedure given in IS 2362.

**ANNEX D**

[Clause 4.2 and Table 1, Sl No. (v)]

**DETERMINATION OF ACIDITY AS ACETIC ACID****D-1 OUTLINE OF THE METHOD**

The material is titrated with standard sodium hydroxide solution to the phenolphthalein end point and from the volume of standard sodium hydroxide solution, used acidity is calculated as acetic acid.

**D-2 REAGENTS****D-2.1 Sodium Hydroxide Solution**

Approximately 0.01 N.

**D-2.2 Hydrochloric Acid**

Approximately 0.01 N.

**D-2.3 Phenolphthalein Indicator**, 0.05 percent (m/m) in isopropyl alcohol.

**D-2.4 Neutralized Isopropyl Alcohol**

Add few drops of phenolphthalein indicator in 50 ml of isopropyl alcohol and add dropwise 0.01 N sodium hydroxide solution till pink colour persists. Neutralize excess of sodium hydroxide by adding 0.01 N

hydrochloric acid until the pink colour has just discharged.

**D-3 PROCEDURE**

Weigh accurately about 100 g of the material to the nearest 0.1 g in a 500 ml conical flask. Add 50 ml of neutralized isopropyl alcohol and few drops of phenolphthalein indicator. Titrate with 0.01 N sodium hydroxide solution using a microburette, until the pink colour persists for 10 seconds.

**D-4 CALCULATION**

$$\text{Acidity (as acetic acid), percent by mass} = \frac{6 \times V \times N}{M}$$

where

$V$  = volume in ml of standard sodium hydroxide solution,

$N$  = normality of standard sodium hydroxide solution, and

$M$  = mass in g of the material taken for the test.

**ANNEX E**

[Clause 4.2 and Table 1, Sl No. (vi)]

**DETERMINATION OF ALDEHYDES AND KETONES (AS CARBONYL GROUP)****E-1 OUTLINE OF THE METHOD**

The aldehydes and ketones present in the material are converted with 2,4-dinitrophenylhydrazine into the corresponding 2,4-dinitrophenylhydrazones. In alkaline medium these produce red colour, which can be determined spectrophotometrically.

**E-2 APPARATUS**

**E-2.1 Spectrophotometer**, with 10 mm path cells.

**E-2.2 Water bath**, controlled at  $60 \pm 1^\circ\text{C}$ .

**E-3 REAGENTS****E-3.1 Carbonyl-Free Methanol**

Reflux 1 000 ml of methanol with 5 g of 2,4-dinitrophenylhydrazine and 2 ml of hydrochloric acid (see IS 265) for 2 hours. Distil off the methanol using a 2 to 3 feet fractionating column. Discard the first 100 ml of distillate. Continue the distillation until approximately 65 percent of the methanol has distilled over.

**E-3.2 2,4-Dinitrophenylhydrazine Solution**

Dissolve 0.10 g of 2,4-dinitrophenylhydrazine in 50 ml of carbonyl-free methanol containing 4 ml of



hydrochloric acid (*see* IS 265) and dilute to 100 ml with water. This solution should be discarded after two weeks.

### E-3.3 Potassium Hydroxide Solution

Dissolve 25 g of potassium hydroxide in 40 ml of distilled water. Cool and dilute to 250 ml with carbonyl-free methanol.

### E-3.4 Methyl IsoButyl Ketone (MIBK)

99.0 percent purity.

### E-3.5 Standard Solution

- Weigh  $0.11 \text{ g} \pm 0.1 \text{ mg}$  of MIBK into 500 ml volumetric flask. Make up to the volume by carbonyl free ethanol.
- Pipette 1.0, 2.0, 5.0, 10.0, 25.0 of the above solution in separate thoroughly cleaned 50 ml volumetric flasks and make up the volume to 30 ml with carbonyl free methanol.
- Calculate amount of carbonyl content present (as C = O) after applying MIBK purity correction, in 2 ml of the above diluted solutions as per following equation:

$$\text{Carbonyl as (C=O) microgram} = 22.4 \times m \times V$$

where

- $m$  = mass of MIBK used above (a), and  
 $V$  = volume of MIBK solution taken for dilution (b) ml.

### E-3.6 Procedure

Pipette 2 ml of each of the five standards E-3.5(b) in five separate 25-ml volumetric flasks. Add 3 ml of carbonyl free methanol to each of above volumetric flasks. Take 5 ml of carbonyl free methanol as blank in the sixth 25-ml volumetric flask. Add 2 ml of 2,4-Dinitrophenylhydrazine solution to each of the above flasks. Heat these flasks in a water bath at  $60 \pm 1^\circ\text{C}$  for 50 minutes. Remove the flasks from the bath and cool to room temperature. Make up the volume to the mark with methanolic KOH solution. Measure the absorbance after  $12 \pm 1$  minutes of the colour development at 480 nm wave length and 10 mm path cell on a spectrophotometer using distilled water as blank. Apply absorbance correction for the blank and plot a calibration chart of mass in microgram against optical density.

Weigh accurately 0.2 g of sample in 25-ml volumetric flask and add 5 ml of carbonyl free methanol and 2 ml of 2,4-Dinitrophenylhydrazine solution and proceed as given in the calibration above. Find out microgram of carbonyl present.

### E-3.7 Calculation

$$\text{Carbonyl compounds (As C=O) \% m/m} = \frac{A}{M \times 10\,000}$$

where

$$M = \text{mass of sample taken for analysis.}$$

## ANNEX F

[Clause 4.2 and Table 1, Sl No. (vii)]

### SULPHURIC ACID COLOUR TEST

#### F-1 OUTLINE OF THE METHOD

The material is treated with concentrated sulphuric acid under specified conditions. Impurities in the sample form coloured compounds by reaction with sulphuric acid and the colour developed is compared with that of a hazen colour standard.

#### F-2 APPARATUS

All apparatus in this test shall be cleaned as follows.

**Precaution** — Rinse each piece of apparatus in warm tap water, dichromate cleaning solution, warm tap water, and methanol; and thoroughly dry with a gently stream of air.

##### F-2.1 Nessler Cylinder

Two, 50 ml capacity (*see* IS 4161).

##### F-2.2 Automatic Burette

10 ml capacity fitted with a TFE-Fluorocarbon stopcock plug.

#### F-3 REAGENT

##### F-3.1 Sulphuric Acid

*See* IS 266.

NOTE — When a new bottle of sulphuric acid is opened for use, its quality should be checked against a previously analysed sample of the material.

#### F-4 PROCEDURE

**F-4.1** Transfer 100 ml of the material to a 250-ml wide-mouth conical flask by means of a measuring cylinder. Add dropwise from a automatic burette 8 ml of the sulphuric acid. Stir the contents of the flask constantly with the aid of a glass stirrer and motor during the addition of acid.

**F-4.2** Cover the mouth of the flask with aluminum foil and place a suitable ring or similar type of mass around the neck of the flask and keep it in the boiling water-bath maintained at  $99$  to  $100^\circ\text{C}$  for a period of  $60 \pm 1$  minutes. Maintain sufficient water in the bath to completely cover the liquid level in the flask.

**F-4.3** Remove the flask from the bath and immediately place in cold tap water. Allow the contents of the flask to cool rapidly to room

temperature. Fill one of the Nessler cylinders to the mark with the treated sample and check the colour as per the standard with IS 8768.

## ANNEX G

[Clause 4.2 and Table 1, Sl No. (viii)]

### DETERMINATION OF PURITY

#### G-1 OUTLINE OF THE METHOD

A small volume of 2-Ethyl Hexan-1-ol is injected into the gas chromatograph containing a capillary column with CP Wax 52 CB as stationary phase.

Quantitative results are obtained by using suitable electronic integrator and internal normalisation method which gives direct concentration of each impurity along with the chromatogram, area, retention time etc.

#### G-2 APPARATUS

Gas chromatograph — Any instrument having a hydrogen flame ionisation detector that may be operated at the conditions given below:

Detector	: Flame Ionisation
Column	: Fused Silica/WCOT
Length	: 50 m
I.D.	: 0.32 mm
Stationary Phase	: CP Wax 52 CB
Film Thickness	: 1.2 micrometre
Injector Type	: Splitter
Temperatures	:
Injection Port	: 250°C
Detector	: 250°C
Oven Initial	: 140°C
Initial Time	: 25 minutes
Programming Rate	: 29 °C/min
Final Temperature	: 200°C
Final Time	: 10 minutes
Carrier Gas	: Nitrogen
Flow Rate	: Vent flow 200 ml/min
Make Up Gas/Flow Rate	: 40 ml/min

FID Hydrogen	: 30 ml/min
Air	: 300 ml/min
Sample Size	: 1.0 microlitre

NOTE — The above Gas Chromatographic conditions are similar to the one given in ASTM D 5008 and are suggestive, however, the packed columns containing Carbowax 20 M or OV<sub>17</sub> as stationary phases may be used provided standardisation/Calibrations are done after setting up chromatographic conditions for the required resolution.

#### G-3 REAGENTS

##### G-3.1 Nitrogen Chromatographic Grade

##### G-3.2 Pure Compound for Calibration

2-Ethyl-4-Methyl Pentanol

#### G-4 PROCEDURE

Prepare a synthetic blend containing known quantities of EMP in EHA. Set the chromatograph and the electronic integrator as required and charge 1 microlitre of sample. Allow the chromatogram to develop. At the end of the run stop the integrator. Run the calibration blend in duplicate. Calculate the internal normalisation factor by dividing the Amount/Area of EMP by the Amount/Area of EHA. Find out the mean factor by averaging.

Charge the sample in the similar manner as given in the calibration above and find out the average area of EMP present in the sample.

#### G-5 CALCULATION

Percent *m/m* EMP = average area of EMP × Factor  
Purity of 2 EHA = 100 – EMP

## ANNEX H

(Clause 7)

### SAMPLING OF 2-ETHYL HEXAN-1-OL

#### H-1 GENERAL REQUIREMENT

**H-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**H-1.1** Sample shall not be taken in an exposed place.

**H-1.2** The sampling instrument shall be clean and dry and shall be made of low or reduced spark generating material.

**H-1.3** The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

**H-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

**H-1.5** The samples shall be placed in suitable, clean, dry and airtight glass containers preferably of amber or blue colour.

**H-1.6** The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

**H-1.7** Rubber stoppers or composition corks shall not be used for closing the sample bottles.

**H-1.8** Sealing wax or other plastic material, if used shall be applied in such a way that it does not contaminate the sample.

**H-1.9** Each sample container shall be protected by cover of oil proof paper, metal foil, viscous or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

**H-1.10** Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture, the batch number, the name of the sampler, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

## H-2 SAMPLING INSTRUMENT

**H-2.0** The following forms of sampling instrument may be used:

- Sampling bottle or can, for taking samples from various depths of large tank; and
- Sampling tube.

### H-2.1 Sampling Bottles or Can

It consists of weighed bottle or metal container with removable stopper or top, to which is attached a light chain (see Fig. 1). The bottle or can is fastened to a

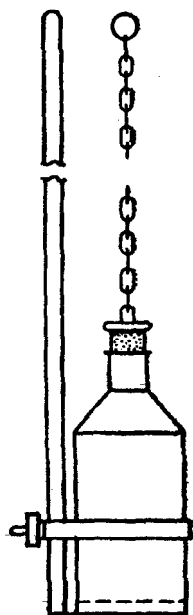


FIG. 1 SAMPLING BOTTLE OR CAN

suitable pole. For taking a sample, it is lowered in the tank to the required depth and the stopper or top is removed by means of the chain for filling the container.

### H-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (see Fig. 2) depending type of container from where the sample is to be drawn. The ends are conical and reach 5 to 10 mm diameter at the tips. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

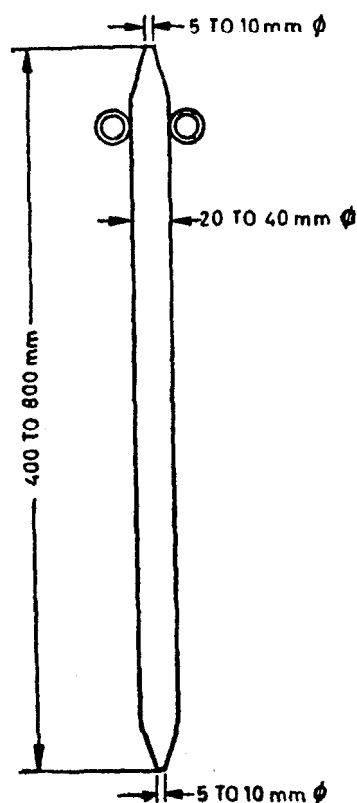


FIG. 2 SAMPLING TUBE

## H-3 SCALE OF SAMPLING

### H-3.1 Lot

In any consignment, all the containers of the same size and drawn from same batch of manufacture shall constitute a lot.

**H-3.2** Tests should be conducted on each lot separately for ascertaining the conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

**Table 2 Scale of Sampling**  
(Clause H-3.2)

Number of Containers in a Lot	Number of Containers to be Selected
Up to 25	3
26 to 100	4
101 " 150	5
151 and above	7

**H-3.3** The containers shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS 4905 may be followed.

#### **H-4 PREPARATION OF THE TEST SAMPLE**

**H-4.1** From each of the containers selected according to **H-3.2** and **H-3.3**, small portions of the material shall be drawn with the help of the sampling instrument from several positions at the upper, middle and bottom layers. These portions shall be mixed together so as to yield a quantity of material not less than 1 000 ml representative of the container.

**H-4.2** From each of these individual samples, an equal quantity of material shall be taken and mixed thoroughly to form a composite sample of about 1 000 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for supplier and third to be used as a referee sample.

**H-4.3** The parts of the composite test sample, obtained according to **H-4.2** shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in **H-1.10**.

**H-4.4** The referee test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

#### **H-5 CRITERIA FOR CONFORMITY**

**H-5.1** The material shall be declared as conforming to the requirements of this specification if the composite sample conforms to all the specified requirements.

**Bureau of Indian Standards**

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**Review of Indian Standards**

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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**Amendments Issued Since Publication**

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